

# PATENT SPECIFICATION

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NO DRAWINGS

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## (54) DYEING PROCESS

(71) We, COURTAULDS LIMITED, a British Company, of 18, Hanover Square, London, W.1, England, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following Statement:—

This invention relates to a dyeing process and in particular to a process for improving colour fastness of dyed nylon fibres and fabrics.

The colour fastness of dyed nylon may be improved by treating the dyed fibres or fabrics with a solution of tannic acid in water at an elevated temperature, preferably with a second treatment in which the treating agent is a solution of potassium antimony tartrate. This is known as the "backtan" treatment. While this process leads to improved colour fastness, in many cases the colour of the dyed material is altered when subsequent washing treatments are required to be carried out at elevated temperatures.

Synthetic "tannings" or "syntans" which are polyhydroxy aromatic compounds cause less discolouration but they do not produce the same colour fastness. It is an object of this invention to provide a process for improving the colour fastness of dyed nylon while retaining substantially unaltered the colour of the dyes.

According to the invention a process for improving the colour fastness of dyed nylon material in the form of fibres or fabric comprises applying to the dyed nylon material an aqueous solution of tannic acid or a syntan, as hereinbefore defined, and applying to the so-treated material a solution of a salt of tin (stannous), bismuth, chromium (chromic) or zinc. The salt solution may be a solution of the sulphate, the chloride or the acetate of the metal.

In the process of the invention tannic acid or syntans may be applied in a bath containing, for example from 2 per cent to 5 per cent of the active material based on the

weight of the fibres or fabric and from 2 per cent to 5 per cent of an acid other than tannic acid or a syntan (for example acetic acid or formic acid) based on the weight of the fibres or fabric (the bath may suitably contain up to about 30 per cent of said acid other than tannic acid or a syntan based on the weight of the solution). The treatment in the tanning bath is carried out at an elevated temperature which may most suitably be about 70°C, for an appreciable time, for example 20 minutes or more. Following the tanning treatment the fibres or fabrics are immersed in a bath containing an aqueous solution of the metal salt for example at a concentration of up to 10 per cent based upon the weight of the fibres or fabric to be treated and also at an elevated temperature for example 70° to 80°C. It is not normally necessary to rinse the fabric between the two treatments. Preferred combinations of treatments include treatments with tannic acid and stannous chloride, bismuth nitrate, chromic acetate or zinc acetate and syntans with zinc acetate or chromic acetate. Suitable syntans include Taninol WR and Nylofixan PE.

Specific combinations of tannic acid or syntan with the metal salt may be found which are of particular value with individual shades.

Fibres and fabrics treated by the process of the invention give good initial fastness and the fastness is substantially unaltered after further treatments at elevated temperature such as by steam setting. Treatment with tannic acid and potassium antimony tartrate gives fibres and fabrics in which the fastness is reduced to an unacceptable level by steam.

The process of the invention is illustrated by the following Examples. In each of the Examples, the figures quoted for dye fastness, expressed by staining of adjacent materials and change of shade, are expressed in units up to 5, which represents the most satisfactory fastness. Satisfactory fastness for "Celon" (Trade Mark) is 4 for staining, and 3 to 4 for change of shade.

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## EXAMPLE 1

A series of experiments was carried out on dyed crimped "Celon" fabric (Nylon 6) in which the fastness of various dyes when treated by the known "backtan" treatment was compared with the fastness obtained by after-treatment according to the process of the invention. The backtan treatment was carried out by immersing the dyed fabric in a first solution containing 2 per cent by weight (based on weight of fabric) of tannic acid and 2 per

cent by weight (based on weight of fabric) of formic acid in water at 70°C for 30 minutes, and then in a solution of potassium antimony tartrate in water, containing 2 per cent of the salt based on the weight of the fabric, at 70°C for 30 minutes.

The fastness of the dye in every case was tested by the ISO 3 standard washing test, in which the fabric was washed in water containing 5 grams of soap per litre and 2 grams of sodium carbonate per litre for 30

TABLE

Tannin/Metal Salt Aftertreatment	Dye employed	No Steam					
		Untreated			Backtanned		
		*cos	soc	sow	cos	soc	sow
2% Tannic Acid/ 2% Stannous chloride	3% Cibalan Brilliant Blue RL	4	2-3	2	4-5	5	5
	3% Eriodin Fast Brown RXL	5	2	4	5	5	5
	3% Nylosan Green E-GL	4-5	2-3	2	5	5	5
2% Tannic Acid/ 2% Chromic acetate	3% Eriodin Fast Brown RXL	5	2	4	5	5	5
	3% Nylosan Navy RNL	5	3	4-5	5	5	5
	3% Polar Red B	5	3	3	5	5	5
2% Tannic Acid/ 2% Zinc acetate	3% Azo Rubinole 3GP	3-4	2	2	4	5	5
	3% Eriodin Fast Brown RXL	5	2	4	5	5	5
	3% Azo Rubinole 3GP	3-4	2	2	4	5	5
3% Taninol WR/ 2% Chromic acetate	3% Eriodin Fast Brown RXL	5	2	4	5	5	5
	3% Lanaperl Fast Red 5B	3-4	2	2	4	5	5
	3% Procilan Dark Blue RS	4	3	5	4-5	4	4
5% Nylofixan PE/ 2% Chromic acetate	3% Azo Rubinole 3GP	3-4	2	2	4	5	5
	3% Azo Rubinole 3GP	3-4	2	2	4	5	5
	3% Azo Rubinole 3GP	3-4	2	2	4	5	5

\*cos = change of shade, soc = staining on "Celon", and sow = staining on wool.

minutes at a temperature of  $60 \pm 2^\circ\text{C}$ . The ratio of the weight of wash liquor to fabric was 50:1, and undyed, scoured, unset crimped "Celon" fabric and wool taffeta fabric were used as adjacent materials.

The experiments are summarised in Table 1, and in each case the fastness of the dyes after steam setting was borderline, satisfactory or unsatisfactory in the fabrics treated by the backtan process, but was entirely satisfactory after treatment by the process of the invention.

#### EXAMPLE 2

Summarised in Table 2 is a series of experiments similar to those in Example 1, in which it was found that the dyes used were significantly discoloured after the ISO standard washing test when the backtan treatment had been applied but that the essential colour of the dye was retained after the washing test when the process of this invention had been used.

Tannin/ Metal Salt Treated			Post Steamed								
			Untreated			Backtanned			Tannin/ Metal Salt Treated		
cos	soc	sow	cos	soc	sow	cos	soc	sow	cos	soc	sow
5	5	5	3-4	2-3	2	4	3-4	4-5	5	5	4-5
5	5	5	5	2	4-5	5	3-4	4-5	5	5	5
5	5	5	4	2-3	2	5	3-4	4-5	5	5	5
5	5	5	5	2	4-5	5	3-4	4-5	5	5	5
5	4-5	5	5	3	4-5	5	3-4	5	5	4-5	5
5	4-5	5	5	3	4	5	3-4	4-5	5	4-5	5
4	4	4-5	3-4	2	2	4-5	2-3	2-3	4	4	4-5
5	4	5	5	2	4-5	5	3-4	4-5	5	4-5	5
4-5	4-5	4	3-4	2	2	4-5	2-3	2-3	4-5	4-5	4-5
4-5	4	5	5	2	4-5	5	3-4	4-5	5	4-5	4-5
4	4	4-5	3-4	2	2	4-5	2-3	2-3	4	4	4-5
5	4-5	5	4-5	2	5	4-5	3	4	5	4-5	4-5
4	4	4-5	3-4	2	2	4-5	2-3	2-3	4	4	4-5
4-5	3-4	4	3-4	2	2	4-5	2-3	2-3	4-5	3-4	4

TABLE 2

Tannin/Metal Salt Aftertreatment	Dye employed	Change of Shade		
		Untreated	Backtanned	Tannin/Metal Salt Treated
2% Tannic Acid/2% Stannous chloride	1% Xylene Fast Yellow RPN	4	3	4
	1% Nylosan Orange E—GL	4-5	3	4
2% Tannic Acid/3% Bismuth nitrate	1% Nylosan Orange E—GL	4-5	3	4
2% Tannic Acid/10% Zinc Acetate	1% Nylosan Orange E—GL	4-5	3	4
3% Taninol WR/10% Zinc acetate	1% Xylene Fast Yellow RPN 1% Nylosan Orange E—GL	4 4-5	3 3	4 4-5

## WHAT WE CLAIM IS:—

1. A process for improving the colour fastness of dyed nylon material in the form of fibres or fabric, comprising applying to the dyed nylon material an aqueous solution of tannic acid or a sytan, as hereinbefore defined, and applying to the so-treated material a solution of a salt of tin (stannous), bismuth, chromium (chromic) or zinc.
2. The process claimed in claim 1, in which the salt solution is a solution of the sulphate, the chloride or the acetate of the metal.
3. The process claimed in claim 1 or 2, in which the tannic acid or sytan is applied in a bath containing from 2 per cent to 5 per cent of the tannic acid or sytan based on the weight of the fibres or fabric and from 2 per cent to 5 per cent of an acid other than tannic acid or a sytan based on the weight of the fibres or fabric.
4. The process claimed in claim 3, in which the bath contains up to 30 per cent of said acid other than tannic acid or a sytan based on the weight of the solution.
5. The process claimed in claim 3 or 4, in which the acid is acetic acid or formic acid.
6. The process claimed in any of the preceding claims, in which the treatment in the tanning bath is carried out at a temperature of about 70°C.
7. The process claimed in claim 6, in which the treatment in the tanning bath is carried out for a period of at least 20 minutes.
8. The process claimed in any of the preceding claims, in which following the tanning treatment the dyed nylon material is immersed in a bath containing an aqueous solution of the metal salt.
9. The process claimed in claim 8, in which the salt solution has a concentration

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of up to 10 per cent based upon the weight of the fibres or fabric.

10. The process claimed in claim 8 or 9, in which the bath containing the metal salt solution has a temperature of from 70° to 80°C.

11. The process claimed in claim 1, in which the dyed nylon material is first treated with an aqueous solution of tannic acid, after which a solution of stannous chloride, bismuth nitrate, chromic acetate or zinc acetate is applied to the so-treated material.

12. The process claimed in claim 1, in which the dyed nylon material is first treated

with an aqueous solution of a syntan, after which a solution of zinc acetate or chromic acetate is applied to the so-treated material.

13. The process claimed in claim 1, substantially as described in either of the foregoing Examples.

14. Dyed nylon material in the form of fibres or fabric when treated by the process claimed in any preceding claim.

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